ADA

ALTERNATIVE FUELS COMPATIBILITY WITH ARMY EQUIPMENT TESTING – EXISTENT GUM

INTERIM REPORT
TFLRF No. 425

by
Scott A. Hutzler

U.S. Army TARDEC Fuels and Lubricants Research Facility
Southwest Research Institute® (SwRI®)
San Antonio, TX

for
U.S. Army TARDEC
Force Projection Technologies
Warren, Michigan

Contract No. W56HZV-09-C-0100 (WD15)

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Gary Bessee

Gary B. Bessee, Director
U.S. Army TARDEC Fuels and Lubricants
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14. ABSTRACT

Many of the materials used for tactical fuel handling equipment were designed for use with petroleum-derived fuels, such as diesel and JP-8, which typically contain 15-25% aromatics. However, emerging synthetic turbine fuels based on iso-paraffinic kerosene (IPK), synthetic paraffinic kerosene (SPK), and Hydroprocessed Esters and Fatty Acids (HEFA) typically contain no aromatics. Many of these fuels have undergone extensive testing and gained approval for use by the Air Force. As these fuels become more widely available and their use extends to ground vehicles and support equipment, their impact on current Army equipment will need to be assessed. ASTM D381 is currently used in specifications for military fuel hoses (MIL-PRF-370J) and collapsible fuel tanks (MIL-PRF-32233). This test method is used to determine if any material in contact with aviation fuels or motor gasolines causes gum contamination. This study addresses various aspects of the ASTM D381 method as a means to determine gum contamination of fuel in contact elastomeric and rubber materials.

15. SUBJECT TERMS

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EXECUTIVE SUMMARY

Many of the materials used for tactical fuel handling equipment were designed for use with petroleum-derived fuels, such as diesel and JP-8, which typically contain 15-25% aromatics. However, emerging synthetic turbine fuels based on iso-paraffinic kerosene (IPK), synthetic paraffinic kerosene (SPK), and Hydroprocessed Esters and Fatty Acids (HEFA) typically contain no aromatics. Many of these fuels have undergone extensive testing and gained approval for use by the Air Force. As these fuels become more widely available and their use extends to ground vehicles and support equipment, their impact on current Army equipment will need to be assessed. ASTM D381 is currently used in specifications for military fuel hoses (MIL-PRF-370J) and collapsible fuel tanks (MIL-PRF-32233). This test method is used to determine if any material in contact with aviation fuels or motor gasolines causes gum contamination. This study addresses various aspects of the ASTM D381 method as a means to determine gum contamination of fuel in contact elastomeric and rubber materials.

As a result of this effort, several required changes to both MIL-PRF-32233 (Collapsible Tanks) and MIL-PRF-370J (Hoses) have been addressed. These changes are needed to bring the terminology of the methods up-to-date with the current ASTM D381 method and clarify specifically how the method should be applied. In addition, suggestions for recommended changes have also been made. None of the proposed changes should invalidate the established acceptance criteria.

FOREWORD/ACKNOWLEDGMENTS

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ACRONYMS AND ABBREVIATIONS

AFRL	Air Force Research Lab
ASTM	American Society for testing and Materials
DIEGME	diethylene glycol monomethyl ether
g	gram
HEFA	Hydroprocessed Esters and Fatty Acids
IPK	Iso-Paraffinic Kerosene
mg	milligram
mL	milliliter
SPK	Synthetic Paraffinic Kerosene
ULSD	Ultra-low Sulfur Diesel
WPAFB	Wright Patterson Air Force Base

1.0 BACKGROUND

Many of the materials used for tactical fuel handling equipment were designed for use with petroleum-derived fuels, such as diesel and JP-8, which typically contain 15-25% aromatics. However, emerging synthetic turbine fuels based on iso-paraffinic kerosene (IPK), synthetic paraffinic kerosene (SPK), and Hydroprocessed Esters and Fatty Acids (HEFA) typically contain no aromatics. Many of these fuels have undergone extensive testing and gained approval for use by the Air Force. As these fuels become more widely available and their use extends to ground vehicles and support equipment, their impact on current Army equipment will need to be assessed.

ASTM D381 is currently used in specifications for military fuel hoses (MIL-PRF-370J) and collapsible fuel tanks (MIL-PRF-32233). This test method is used to determine if any material in contact with aviation fuels or motor gasolines causes gum contamination. Research is needed to determine if this test method is appropriate for this purpose given the variety of emerging fuels.

2.0 OBJECTIVES

The specific objective for this effort was as follows:

- Determine if ASTM D381 is a suitable test method for determining gum contamination of fuels in contact with current tank and hose material.

This effort can be approached from two angles:

- effect on the fuel (i.e. gum contamination)
- effect on the material (e.g. reduction in tensile strength)

This study will attempt to weigh-in on both issues and show that ASTM D381 is a valuable method and should remain a part of the performance specifications.

3.0 APPROACH

1) Identify the elastomeric materials used in current fuel hoses and collapsible fuel tanks associated with tactical fuel handling equipment.
2) Analyze the extractable content of the elastomeric material to verify that the appropriate temperature at which the test should be performed.
3) Evaluate ASTM D381 as a means to determine the extractable content of the elastomeric material. Consider various aspects of the procedure such as:
    a. evaporation time
    b. evaporation temperature
    c. solvent choice for solvent washed gums
    d. amount of material to extract
    e. number of replicates
    f. surface-area for the extraction step
4) Determine the appropriate testing medium by performing a comparison of extraction efficiencies using ASTM reference fuels, current fuels (aviation, diesel, gasoline), emerging fuels (SPK, IPK, HEFA), and petroleum/synthetic blends.

4.0 METHODS, SPECIFICATIONS, & PROCEDURES

Current versions of the primary performance specifications and test protocols were obtained:

- MIL-PRF-32233 - Performance Specification: Tanks, Collapsible, 3,000, 10,000, 20,000, 50,000, & 210,000 U.S. Gallons, Fuel (20 Dec 2006)
- MIL-PRF-370J - Performance Specification: Hose And Hose Assemblies, Nonmetallic: Elastomeric, Liquid Fuel (07 May 2002)
- ASTM D381-09 Standard Test Method for Gum Content in Fuels by Jet Evaporation
- ASTM D471-10 Standard Test Method for Rubber Property—Effect of Liquids

The methods for determining gum content in MIL-PRF-32233 and MIL-PRF-370J are adaptations of ASTM D381 and call for reference fluids defined in ASTM D471.

4.1 MIL-PRF-32233, COLLAPSIBLE TANKS

The gum content method as defined in MIL_PRF-32233 is as follows:

> 4.5.2.2 Gum Content. Cut a 0.2-ounce specimen of each tank interior coating compound into approximately 0.0625-inch squares and place in a flask containing 8.5 fluid ounces of Reference Fuel D of ASTM D 471 and allow to stand for 48 hours at 73 ±5 °F. Decant and filter the contaminated fluid through Whatman 41H filter paper or equal. Determine the unwashed gum content of the filtrate in accordance with ASTM D 381, procedures 11.1 through 11.12 using the air jet or steam jet vaporizing medium (which is appropriate for the fuel) and an evaporation time of 45 minutes. Using the same samples after completing the unwashed gum content test above, determine the existent gum in accordance with ASTM D 381, procedures 11.1 through 11.12. A minimum of three specimens shall be used. Nonconformance to Table I shall constitute failure of the test. This test shall be repeated with Reference Fuel B, Aviation Gasoline per ASTM D 910 Grade 100LL, JP-8, and DL2.

4.1.1 MIL-PRF-32233 Performance Characteristics

The acceptance criteria for gum contamination in MIL-PRF-32233 is found in Table 1. This method has maximum limits for both washed and unwashed gums.

Table 1. MIL-PRF-32233 Table I Characteristics of Tank Material Interior/Exterior

Test Property	Requirements	Test Reference		Application
		ASTM	Para	
Fuel Contamination: Unwashed gum - Existent gum -	20 mg/100 mL (max) 5 mg/100 mL (max)	D381	4.5.2.2	Internal

4.2 MIL-PRF-370J, HOSES

The gum content method for hoses is defined in MIL-PRF-370J. It follows ASTM D381 much closer than the tank method but is a much longer test at 168 hours soak time.

4.3.6.6 Existent gum. A hose specimen 14-in. long shall be cut from the selected test item. One end of the hose shall be stoppered with a clean non-corrosive plug, and secured with a suitable clamp. The hose shall be filled to within 2 in. of the open end with reference fuel B as specified in ASTM D471, and plugged in a manner similar to the other end. A sample of the fuel shall be retained for later use. This hose section shall be stored in a vertical position for 168 hours at a temperature of 100 ± 4°F. At the end of each 24-hour period, the fuel shall be agitated for 5 minutes by moving the specimen from the vertical to the horizontal and back to the vertical position at a rate of 2 cycles per minute. On completion of the 168-hour period, the fuel shall be agitated, as before, for 5 minutes and immediately removed. The removed fuel shall be tested for existent gum IAW the air-jet solvent wash method of ASTM D381. The retained fuel sample shall be tested at the same time and with the same method. The existent gum from the original fuel sample shall be subtracted from the existent gum obtained from the fuel removed from the test hose. Nonconformance to Table III shall constitute failure of this test.

4.2.1 MIL-PRF-370J Performance Criteria

The performance criteria for hoses is given in Table III of MIL-PRF-370J and are shown in Table 2.

Table 2. MIL-PRF-370J Table III Hose Physical and Chemical Properties

Physical properties	Hose Type*				Requirements	Test Method	
	A	B	C	D		ASTM	Test
Existent gum	X	X		X	20 mg / 100 mL	D381	4.3.6.6
			X		6 mg / 100 mL	D471	

* See MIL-PRF-370J for hose types

4.3 ASTM D381 GUM CONTENT

ASTM D381 is used to determine the gum content of aviation gasolines and turbine fuels by jet evaporation.

Summary of Test Method (Excerpt from ASTM D381-09)

4. Summary of Test Method

4.1 When testing either aviation or motor gasoline, a 50 ± 0.5 mL quantity of fuel is evaporated under controlled conditions of temperature and flow of air. When testing aviation turbine fuel, a 50 ± 0.5 mL quantity of fuel is evaporated under controlled conditions of temperature and flow of steam. For aviation gasoline and aviation turbine fuel, the resulting residue is weighed and reported as milligrams per 100 mL. For motor gasoline, the residue is weighed before and after extracting with heptane and the results reported as milligrams per 100 mL.

5.0 TEST MATERIALS

The following sections describe the materials used in this study.

5.1 COLLAPSIBLE TANK MATERIAL

The collapsible tank material used in this study was a polyurethane coated fabric. This material was provided free of charge by a vendor who simply wished to remain anonymous. This vendor is a major supplier of coated fabric and several tanks on the market are believed to be constructed of this material.

MIL-PRF-32233 specifically discusses the use of the “interior” coating compound of the tanks but does not address the use of the actual coated fabric. To our knowledge the interior and exterior coating of current collapsible tanks are the same. This calls into question whether a special run of elastomeric material, sans fabric, should be prepared specifically for first article testing. The soak tests are performed on a specific mass of material that is already finely divided to maximize surface area. The penetration of the test fluid into the fabric would simply increase the exposed surface area of the tank material. A few experiments were conducted to that end to see if surface area was a major factor in extracted gum content. Those results are described below.

5.2 HOSE MATERIAL

The original plan was to acquire a hose that meets MIL-PRF-370J to serve as a baseline for the soak tests. However, it was quickly discovered that obtaining such a hose was impractical for reasons of time, cost, and quantity (2 months lead-time, $5K, 500ft minimum). As an alternative, the following petroleum hose from Goodyear was chosen. This hose is not specifically recommended for jet fuel. Therefore, it may yield less-than-optimal results with the test fluids selected for this study as they are primarily alternative aviation fuels. Nevertheless, the hose should provide an adequate baseline for comparing fuels and experimental variables.

Plicord® Flexwing® Petroleum

APPLICATION:

For use in tank truck and in-plant operations to transfer gasoline, oil, ethanol blends and other petroleum base products up to 50% aromatic content. It is designed for pressure, gravity flow, or full-suction service.

CONSTRUCTION:

TUBE:	Nitrile synthetic rubber RMA Class A (High Oil Resistance)
COVER:	Red Chemivic™ (white spiral stripe) synthetic rubber (oil resistant); smooth cover; wrapped finish
REINFORCEMENT:	Spiral-plied synthetic fabric with wire helix
TEMPERATURE:	-35°F to 200°F (-37°C to 93°C)

5.3 TEST FLUIDS

The test fluids used in this study are shown in Table 3. The goal was to incorporate as many of the emerging alternative aviation fuels as possible for comparison to the petroleum-based fuels and the solvent-based fuel surrogates commonly referenced in the military specifications. The synthetic fuels in this study were provided by the Air Force Research Laboratory (AFRL) at Wright Patterson Air Force Base. (WPAFB). With the exception of the Rentech fuel, a detailed discussion of the fuels used in this study has already been published[1]. A complete report of the Rentech fuel is pending and should be released by AFRL in mid 2012.

Portions of the fuels were clay-treated to remove trace additives and water and to address the possibility of removing inherent gums from the neat fuels which affect the final gum result. As shown in the table, various 50/50 volumetric blends were prepared to study their impact on gum extraction.

The solvent-based fuel surrogates were prepared according to ASTM D471 and are used in this study in lieu of aviation or motor gasolines (which would be expected to behave in a similar manner). A 90/10 isooctane/toluene blend was proposed as a surrogate to mimic the expected low aromatic content of the 50/50 synthetic/petroleum blends.

Table 3. Test Fluids

SwRI Sample ID	Description	Comments
Neat Fuels		
CL09-0268	Sasol IPK (neat)	as received from AFRL
CL10-0326	R-8 HEFA SPK (neat)	as received from AFRL
CL12-3318	Jet A (neat)	purchased from Valero
CL11-3131	Rentech FT-SPK (neat)	as received from AFRL
CL11-3118	Camelina HEFA SPK (neat)	as received from AFRL
CL11-3117	Tallow HEFA SPK (neat)	as received from AFRL
CL11-3107	JP-8	prepared from CL11-3100*
CL11-3108	ULSD	local filling station
Clay-Treated Fuels		
CL11-2946	CT Sasol IPK	clay treated CL09-0268
CL11-2947	CT R-8	clay treated CL10-0326
CL11-3100	CT Jet A	clay treated CL12-3318
CL11-3147	CT Rentech	clay treated CL11-3131
CL11-3148	CT Camelina	clay treated CL11-3118
CL11-3149	CT Tallow	clay treated CL11-3117
50/50 Blends (additized to JP-8)		
CL11-3135	CT IPK / JP-8	50/50 volumetric blends: clay-treated synthetic / clay-treated Jet A + JP-8 additives*
CL11-3150	CT R-8 / JP-8	
CL11-3153	CT Rentech / JP-8	
CL11-3154	CT Camelina / JP-8	
CL11-3155	CT Tallow / JP-8	

Table 3. Test Fluids

SwRI Sample ID	Description	Comments
50/50 Blends		
CL12-3313	IPK CL09-0268 / Jet A	50/50 volumetric blends: synthetic / Jet A (CL12-3318) as received
CL12-3314	R-8 CL10-0326 / Jet A	
CL12-3315	Camelina CL11-3118 / Jet A	
CL12-3316	Tallow CL11-3117 / Jet A	
CL12-3317	Rentech CL11-3131 / Jet A	
Solvent Blends (Fuel Surrogates)		
CL12-3310	Fuel B 70/30 isooctane / toluene	
CL12-3311	Fuel D 60/40 isooctane / toluene	
CL12-3312	Fuel XX 90/10 isooctane / toluene	proposed surrogate to mimic low aromatic content blends

*JP-8 blends: ~1 mg/L Stadis 450, 15 mg/L DCI-4A, 0.15 vol% DIEGME

6.0 RESULTS AND DISCUSSIONS

6.1 ACCEPTANCE CRITERIA

A major obstacle when considering a modification to an existing procedure is the effect on the established acceptance criteria. For that reason, we must be careful that procedural changes will not affect these criteria unless we are in a position to generate enough data to create new acceptance criteria. Generating new acceptance criteria is likely beyond the scope of this effort because it would require a large array of materials and fuels. The focus of the work will be to verify that D381 is a suitable method while simultaneously verifying that alternative aviation fuels behave in a similar manner to petroleum-based fuels relative to gum extraction.

6.2 EVAPORATION TEMPERATURES - FUEL VOLATILITY

ASTM D381 provides for two evaporation temperatures depending on the fluid being tested. Aviation and motor gasolines use a cooler air jet temperature giving a well temperature of 150-160°C. Aviation turbine fuels use steam as a vaporizing medium giving a well temperature of 229-235°C. Given the similarity of the alternative aviation fuels to the petroleum-based turbine fuels, it would be expected that those would also use steam evaporation. The lighter nature of the solvent blends requires air to be used as the vaporizing medium. Although D381 makes no specific provision for heavier fuels, like ULSD, it is presumed that steam would be used where called for in a military specification. However, given that diesel fuels have a higher boiling range, it is expected that the residues might be significantly higher and may need to be corrected for comparison to the performance specification criteria.

The boiling ranges of the test fluids, measured using simulated distillation (ASTM D2887), are shown in Figure 1 and tabulated in Table 4 through Table 8. Figure 1 confirms that the alternative aviation fuels and their blends are not significantly different in boiling characteristic so the use of steam as the vaporizing medium should give comparable results to petroleum-based fluids. The air temperature is more than adequate to evaporate the solvent blends.

In Figure 2 (Table 9), high temperature simulated distillation was used to obtain the boiling range of several residues collected following an elastomer soak test (2g material, ~0.0625” squares, 48 hour soak, room temperature). The solvents were removed under the air jet temperature for 30 minutes and the residues collected in carbon disulfide. This data shows that the residues are significantly heavier than aviation fuel and solvent blends and are therefore not expected to be lost during the evaporation process.

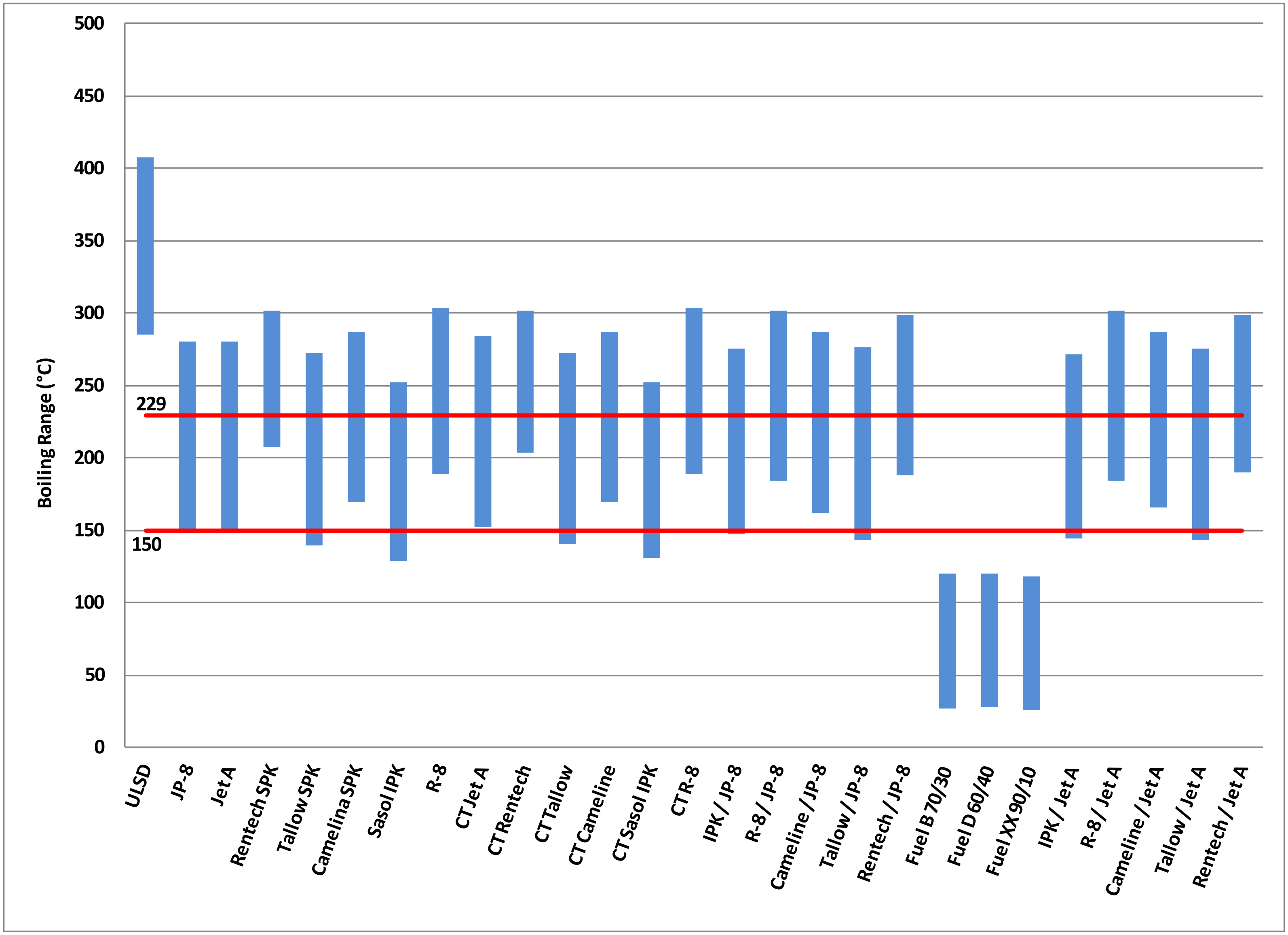

Figure 1. Comparison of Test Fluid Boiling Ranges

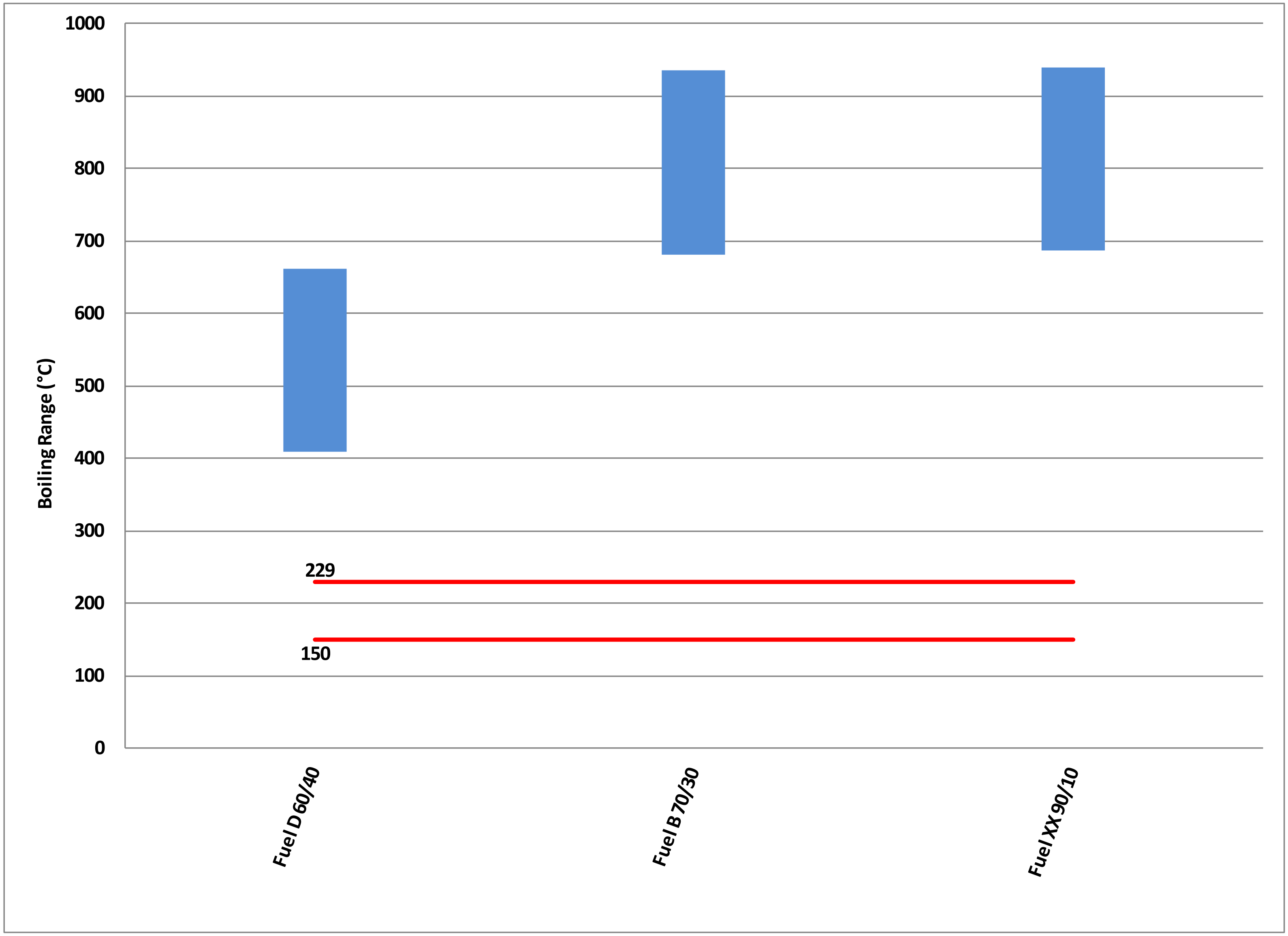

Figure 2. High Temperature Simulated Distillation of Gum Residues

Table 4. Simulated Distillation (°C) – Neat Fuels

Sample Number	CL11-3108	CL11-3107	CL12-3318	CL11-3131	CL11-3117	CL11-3118	CL09-0268	CL10-0326
Description	ULSD	JP-8	Jet A	Rentech SPK	Tallow SPK	Camelina SPK	Sasol IPK	R-8 SPK
IBP	122.8	132.5	131.2	94.3	133.1	118.0	123.2	115.5
5%	168.1	153.9	152.9	126.7	143.9	137.0	139.0	142.4
10%	183.9	162.1	161.4	142.7	156.8	143.0	146.5	151.9
15%	196.8	167.1	166.5	150.9	165.1	144.9	154.3	164.8
20%	209.6	170.7	170.1	161.6	168.4	152.1	156.9	168.7
25%	219.5	174.1	173.5	167.5	178.9	158.2	161.2	179.1
30%	230.4	175.5	175.0	178.3	186.7	164.1	166.5	187.5
35%	239.1	177.8	177.1	187.5	190.8	166.7	170.1	194.3
40%	248.4	180.9	180.3	196.2	199.7	168.9	173.4	200.9
45%	255.6	185.2	184.4	203.9	206.4	177.0	177.0	208.3
50%	264.0	188.7	188.2	210.9	210.8	181.4	179.6	215.8
55%	271.4	193.3	192.6	219.8	218.5	187.6	182.7	221.9
60%	279.8	196.9	196.3	228.1	225.4	194.0	187.0	229.3
65%	287.8	198.3	197.8	236.4	231.6	201.8	191.4	237.5
70%	296.0	202.9	202.3	244.9	240.1	209.9	194.4	246.3
75%	303.5	209.3	208.7	253.0	247.1	219.4	199.0	255.6
80%	312.9	216.1	215.5	260.4	251.9	228.4	204.8	263.7
85%	322.3	220.3	219.7	268.4	255.8	239.1	211.5	270.3
90%	334.3	230.7	230.2	276.4	258.7	253.2	220.2	276.0
95%	352.9	242.6	242.0	285.2	263.3	265.6	230.2	286.1
FBP	407.5	280.5	280.2	302.0	272.7	287.5	252.5	304.1

Table 5. Simulated Distillation (°C) – Clay-Treated Fuels

Sample Number	CL11-3148	CL11-2946	CL11-3147	CL11-3149	CL11-2947	CL11-3100
Description	**CT Camelina**	**CT Sasol IPK**	**CT Rentech**	**CT Tallow**	**CT R-8**	**CT Jet A**
IBP	118.1	122.3	97.6	132.7	114.8	131.9
5%	137.5	138.7	127.0	143.8	142.2	153.6
10%	142.9	146.3	142.6	156.7	151.7	162.0
15%	144.8	154.1	151.0	165.0	164.6	167.0
20%	152.1	156.7	163.0	168.2	168.6	170.7
25%	158.3	161.0	167.7	178.8	179.0	174.1
30%	164.3	166.3	178.8	186.6	187.3	175.5
35%	166.6	170.0	187.7	190.7	194.4	177.8
40%	169.1	173.2	196.4	199.6	200.9	180.9
45%	177.2	176.8	204.8	206.4	208.2	185.1
50%	181.6	179.5	211.4	210.7	215.8	188.7
55%	187.7	182.5	220.1	218.5	222.1	193.3
60%	195.0	186.9	228.3	225.2	229.3	196.9
65%	202.1	191.2	236.5	231.4	237.6	198.3
70%	210.0	194.2	245.0	240.0	246.3	202.9
75%	219.5	198.8	253.1	247.0	255.6	209.3
80%	228.5	204.7	260.5	251.9	263.7	216.2
85%	239.2	211.3	268.5	255.7	270.2	220.4
90%	253.3	220.1	276.3	258.7	276.0	230.8
95%	265.5	230.1	285.1	263.3	286.1	243.0
FBP	287.6	252.6	301.6	272.9	304.1	284.0

Table 6. Simulated Distillation (°C) – JP-8 Blends

Sample Number	CL11-3135	CL11-3150	CL11-3154	CL11-3155	CL11-3153
Description	CT IPK / JP-8	CT R-8 / JP-8	CT Camelina / JP-8	CT Tallow / JP-8	CT Rentech / JP-8
IBP	128.5	117.8	124.8	133.1	110.8
5%	144.7	148.9	142.8	150.8	142.1
10%	154.2	159.0	151.0	159.6	151.8
15%	159.0	166.0	158.1	166.2	162.6
20%	165.2	170.4	164.6	170.1	167.1
25%	168.7	174.3	167.2	174.5	172.9
30%	172.6	178.3	171.3	178.4	175.1
35%	174.7	182.6	174.8	182.2	179.9
40%	177.4	187.9	178.3	187.8	185.8
45%	180.3	193.3	181.6	192.3	190.0
50%	184.1	196.9	186.9	197.0	196.1
55%	188.1	201.8	190.5	201.1	199.6
60%	192.3	208.4	196.5	207.7	207.0
65%	195.9	215.5	200.1	213.3	213.7
70%	198.6	220.6	207.3	218.9	219.4
75%	204.3	229.4	214.2	227.2	228.6
80%	210.4	238.2	220.0	235.8	237.1
85%	216.8	251.5	229.7	245.8	248.4
90%	226.0	264.2	240.5	253.9	262.1
95%	236.6	276.4	257.5	260.0	276.8
FBP	275.5	301.9	286.9	276.9	298.4

Table 7. Simulated Distillation (°C) – Jet A Blends

Sample Number	CL12-3313	CL12-3314	CL12-3315	CL12-3316	CL12-3317
Description	**IPK / Jet A**	**R-8 / Jet A**	**Camelina / Jet A**	**Tallow / Jet A**	**Rentech / Jet A**
IBP	127.5	117.2	121.2	132.6	108.1
5%	144.4	146.5	142.4	150.4	141.8
10%	154.0	158.4	150.1	159.1	151.5
15%	158.7	165.8	157.3	165.8	161.9
20%	164.9	169.8	163.7	169.5	166.9
25%	168.5	174.3	166.7	174.2	172.6
30%	172.3	177.7	170.2	177.8	175.0
35%	174.7	182.1	174.4	181.7	179.7
40%	177.2	187.7	177.4	187.3	185.5
45%	180.2	192.6	180.9	191.6	189.7
50%	183.6	196.9	186.2	196.7	195.9
55%	187.9	201.4	189.9	200.6	199.4
60%	192.0	208.2	196.0	207.2	206.8
65%	195.7	215.2	199.4	212.6	213.4
70%	198.4	220.2	206.6	218.4	219.2
75%	203.9	229.2	213.4	226.7	228.4
80%	210.2	237.8	219.5	235.3	236.9
85%	216.8	250.7	229.2	245.4	248.3
90%	225.6	264.1	239.9	253.5	262.0
95%	236.4	276.3	257.0	259.7	276.6
FBP	271.8	301.8	286.7	275.8	298.5

Table 8. Simulated Distillation (°C) – Solvent Blends

Sample Number	CL12-3310	CL12-3311	CL12-3312
Description	**Fuel B 70/30**	**Fuel D 60/40**	**Fuel XX 90/10**
IBP	92.7	92.6	92.6
5%	94.3	94.2	94.3
10%	95.0	94.9	95.0
15%	95.5	95.4	95.5
20%	95.9	95.8	95.8
25%	96.2	96.1	96.2
30%	96.5	96.4	96.5
35%	96.7	96.7	96.7
40%	97.0	96.9	97.0
45%	97.2	97.2	97.2
50%	97.4	100.4	97.4
55%	97.6	117.0	97.6
60%	97.9	117.6	97.8
65%	116.4	118.0	97.9
70%	117.4	118.4	98.1
75%	118.0	118.7	98.3
80%	118.4	119.0	98.4
85%	118.7	119.2	98.7
90%	119.0	119.5	116.3
95%	119.3	119.7	117.5
FBP	119.9	120.2	118.4

Table 9. High Temperature Simulated Distillation (°C) – Gum Extracts

Sample Number	CL12-3353	CL12-3354	CL12-3355
Description	**Fuel D 60/40**	**Fuel B 70/30**	**Fuel XX 90/10**
IBP	251.7	254.5	252.4
5%	289.3	392.8	380.5
10%	354.7	405.7	405.0
15%	405.4	406.6	405.9
20%	406.9	407.4	406.6
25%	407.8	426.6	407.5
30%	410.4	429.7	426.8
35%	429.0	449.7	437.2
40%	438.2	479.1	452.2
45%	451.8	513.4	501.7
50%	485.8	530.5	519.2
55%	513.3	550.1	535.9
60%	529.5	580.3	558.1
65%	546.1	607.6	595.8
70%	568.1	633.7	629.5
75%	595.0	637.6	637.3
80%	612.5	643.3	644.5
85%	634.7	686.3	728.4
90%	638.2	769.8	790.8
95%	641.7	849.5	863.4
FBP	661.7	935.7	940.0

6.3 BASELINE GUM MEASUREMENTS

The baseline values for the test fluids are shown in Table 10. For the solvent blends, the washed gums are also shown. In all cases, other than ULSD, the inherent existent/unwashed gum content was found to be≤1 mg/100 mL. Since the inherent values are so low, it is difficult to conclude whether the clay-treating had any effect on the gum content. Although the values are generally low, results can vary in real-world samples – up to 30% of the limit criteria stated in the performance specifications. Therefore, when performing soak tests the baseline gum content of the test fluid should be removed by subtraction from the final gum content.

Table 10. Baseline Gum Measurements

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1	Run 2	Average	Run 1	Run 2	Average
CL09-0268	Sasol IPK (neat)	<1	<1	<1			
CL10-0326	R-8 HEFA SPK (neat)	<1	<1	<1			
CL12-3318	Jet A (neat)	1	1	1			
CL11-3131	Rentech FT-SPK (neat)	<1	<1	<1			
CL11-3118	Camelina HEFA SPK (neat)	<1	<1	<1			
CL11-3117	Tallow HEFA SPK (neat)	<1	<1	<1			
CL11-3107	JP-8	1	1	1			
CL11-3108	ULSD	38	38	38			
CL11-2946	CT Sasol IPK	<1	<1	<1			
CL11-2947	CT R-8	<1	<1	<1			
CL11-3100	CT Jet A	<1	<1	<1			
CL11-3147	CT Rentech	<1	<1	<1			
CL11-3148	CT Camelina	<1	<1	<1			
CL11-3149	CT Tallow	<1	<1	<1			
CL11-3135	CT IPK / JP-8	<1	<1	<1			
CL11-3150	CT R-8 / JP-8	<1	1	<1			
CL11-3153	CT Rentech / JP-8	<1	1	<1			
CL11-3154	CT Camelina / JP-8	<1	<1	<1			
CL11-3155	CT Tallow / JP-8	1	1	1			
CL12-3313	IPK CL09-0268 / Jet A	1	1	1			
CL12-3314	R-8 CL10-0326 / Jet A	<1	<1	<1			
CL12-3315	Camelina CL11-3118 / Jet A	<1	<1	<1			
CL12-3316	Tallow CL11-3117 / Jet A	1	1	1			
CL12-3317	Rentech CL11-3131 / Jet A	1	1	1			
CL12-3310	Fuel B 70/30 isooctane / toluene	<1	<1	<1	<0.5	<0.5	<0.5
CL12-3311	Fuel D 60/40 isooctane / toluene	<1	<1	<1	<0.5	<0.5	<0.5
CL12-3312	Fuel XX 90/10 isooctane / toluene	<1	<1	<1	<0.5	<0.5	<0.5

6.4 POLYURETHANE SOAK TESTS

Soak tests on the polyurethane material were performed according to MIL-PRF-32233. The prescribed conditions for the procedure are as follows:

- 0.2-ounce (5.67g) specimen

- ~0.0625-inch squares
- 8.5 fluid ounces (251.4 mL) of Reference Fuel D
- 48 hours at 73 ±5 °F
- Filter through Whatman 41H filter paper
- Air or steam as appropriate
- 45 min evaporation time
- Unwashed and/or washed content
- Three replicates (A, B, C)

With few exceptions, the repeatability between runs of the same bottle and between replicates on the same fuel are remarkably good (Table 11). This data appears to show an emerging trend linking increasing gum content to an increase in aromatic content. Running three replicates is preferred because occasionally an anomalous result will be obtained. In addition, running duplicate samples from the same flask is also advisable to verify repeatability.

Table 11. Polyurethane Soak Tests – MIL-PRF-32233

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1	Run 2	Average	Run 1	Run 2	Average
CL11-3318A	Jet A	2	2	2			
CL11-3318B		3	3	3			
CL11-3318C		2	2	2			
			Average	**2.3**			
CL11-3107A	JP-8	7	7	7			
CL11-3107B		7	7	7			
CL11-3107C		5	6	6			
			Average	**6.7**			
CL11-3108A	ULSD (neat)	13	13	13			
CL11-3108B		15	15	15			
CL11-3108C		14	15	14			
			Average	**14.0**			
CL09-0268A	Sasol IPK (neat)	1	1	1			
CL09-0268B		1	2	1			
CL09-0268C		1	1	1			
			Average	**1.0**			
CL10-0326A	R-8 (neat)	3	3	3			
CL10-0326B		2	2	2			
CL10-0326C		2	2	2			
			Average	**2.3**			
CL11-2946A	CT Sasol IPK	<1	<1	<1			
CL11-2946B		<1	1	<1			
CL11-2946C		1	1	1			
			Average	**0.3**			
CL11-2947A	CT R-8	1	1	1			
CL11-2947B		1	1	1			
CL11-2947C		1	1	1			
			Average	**1.0**			
CL11-3147A	CT Rentech	>1	1	<1			
CL11-3147B		1	1	1			
CL11-3147C		1	1	1			
			Average	**1.0**			

Table 11. Polyurethane Soak Tests – MIL-PRF-32233

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1	Run 2	Average	Run 1	Run 2	Average
CL11-3148A	CT Camelina	1	1	1			
CL11-3148B		1	1	1			
			Average	**1.0**			
CL11-3135A	CT IPK / JP8	3	3	3			
CL11-3135B		5	5	5			
			Average	**4.0**			
CL11-3150A	CT R-8 / JP-8	2	2	2			
CL11-3150B		2	2	2			
			Average	**2.0**			
CL11-3153A	CT Rentech / JP-8	10	10	10			
CL11-3153B		8	8	8			
			Average	**9.0**			
CL11-3154A	CT Camelina / JP8	5	6	5			
CL11-3154B		5	5	5			
			Average	**5.0**			
CL11-3155A	CT Tallow / JP-8	3	3	3			
CL11-3155B		4	4	4			
			Average	**3.5**			
CL11-3310A	Fuel B 70/30 Isooctane/Toluene	17	17	17	<0.5	<0.5	<0.5
CL11-3310B		14	14	14	<0.5	<0.5	<0.5
			Average	**15.5**			
CL11-3311A	Fuel D 60/40 Isooctane/Toluene	29	29	29	<0.5	<0.5	<0.5
CL11-3311B		20	20	20	<0.5	<0.5	<0.5
			Average	**24.5**			
CL11-3312A	Fuel XX 90/10 Isooctane/Toluene	15	15	15	1.0	1.0	1.0
CL11-3312B		14	14	14	2.0	2.0	2.0
			Average	**14.5**			

6.5 HOSE SOAK TESTS

Soak tests on the hose material were performed according to MIL-PRF-370J. This specification uses only Fuel B to find the washed gum content under air-jet evaporation. This study extended the testing to include the other fluids using air or steam-jet evaporation as appropriate to find the existent/unwashed or washed gum content. The hoses, as delivered, were found to be exceptionally dirty on the inside. Therefore, a filtration step was added to the procedure like that used in MIL-PRF-32233 to remove large debris after the soak period was complete.

The prescribed conditions for the procedure are below.

- 14-inch section of hose
- Reference Fuel B
- 168 hours at 100 ±4 °F (daily agitation)
- ***Filter through Whatman 41H filter paper (added)***

- Air or steam-jet evaporation
- Existent/Unwashed or washed gum content

The extraction of gum content from the hose was found to be excessive in most of the fluids (Table 12). The large values for Fuel D were the result of a large, solid residue mass that did not evaporate but was removed by the solvent washing. Most of the turbine fuels gave similar gum contents. This hose would not have passed with a gum content of 35 mg/100 mL for Fuel B.

Table 12. Hose Tests – MIL-PRF-370J

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1*	Run 2*	Average*	Run 1	Run 2	Average
CL11-3107	JP-8	48 (47)	48 (47)	48 (47)			
CL12-3318	Jet A	42 (41)	42 (41)	42 (41)			
CL11-3108	ULSD	124 (86)	125 (87)	124 (86)			
CL11-3117	Tallow (neat)	40	41	40			
CL11-3118	Camelina (neat)	34	34	34			
CL11-3131	Rentech (neat)	37	37	37			
CL11-2946	CT Sasol IPK	28	28	28			
CL11-2947	CT R-8	31	31	31			
CL11-3147	CT Rentech	35	35	35			
CL11-3148	CT Camelina	35	35	35			
CL11-3149	CT Tallow	34	34	34			
CL11-3135	CT IPK / JP-8	32	32	32			
CL11-3150	CT R-8 / JP-8	30	31	30			
CL11-3153	CT Rentech / JP-8	24	24	24			
CL11-3154	CT Camelina / JP-8	57	56	56			
CL11-3155	CT Tallow / JP-8	55 (54)	55 (54)	55 (54)			
CL11-3311	Fuel D 60/40 Isooctane/Toluene	1743	1743	1743	<0.5	<0.5	<0.5
CL11-3310	Fuel B 70/30 Isooctane/Toluene	92	92	92	35.0	35.0	35.0
CL11-3312	Fuel XX 90/10 Isooctane/Toluene	25	26	25	10.5	9.5	10.0

*The values in parentheses are corrected for the baseline values of the fluid

6.6 ROOM TEMPERATURE HOSE SOAK TESTS

Selected fluids from the previous hose tests were repeated under the same procedure except that the hoses were held at room temperature rather than 100°F for 1-week. The results obtained from this testing (Table 13) show that the temperature does influence the amount of gum extracted from the hose. These results were substantially less that their 100°F counterparts.

Table 13. Hose Tests at Room Temperature – MIL-PRF-370J

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1*	Run 2*	Average*	Run 1	Run 2	Average
CL11-3107	JP-8	30 (29)	30 (29)	30 (29)			
CL11-3108	ULSD (neat)	62 (24)	62 (24)	62 (24)			

CL11-3135	CT IPK / JP8	25	25	25			
CL11-3153	CT Rentech / JP-8	19	19	19			
CL11-3154	CT Camelina / JP8	26 (25)	26 (25)	26 (25)			

*The values in parentheses are corrected for the baseline values of the fluid

6.7 SPECIMEN SIZE

Specimen preparation is one of the primary challenges of MIL-PRF-32233. The procedure requires that square specimens of approximately 0.0625” (1/16”) be cut from a sheet of the tank material. From experience, most operators are unable to cut such small specimens with accuracy. The coated fabric used in this effort would not remain intact when cut to such small dimensions. Sheets of elastomer are often stiff and difficult to cut. On average, the typical operator seems to achieve specimens sizes between 1/16” and 1/8”. To test whether specimen size (and thus surface area) has an effect on the extracted gum content, three samples of varying dimension (squares of 0.25”, 0.5”, and 1.0”) were soaked in Fuel D and analyzed per the MIL-PRF-32233 procedure (Table 14).

Although this is by no means an exhaustive study, the results suggests that the extracted gum content is similar regardless of the specimen size. The 1.0” specimens gave slightly higher results but are probably commensurate with the slight increase in sample size. This is at least encouraging that extreme accuracy may not be necessary when cutting the material.

Table 14. Specimen Size – MIL-PRF-32233

SwRI Sample ID	Description	Existent / Unwashed Gums mg/100mL			Washed Gums mg/100mL		
		Run 1*	Run 2*	Average*	Run 1	Run 2	Average
CL11-3311	Fuel D 60/40 isooctane / toluene 5.66g, 0.25" squares	20	20	20	<0.5	<0.5	<0.5
CL11-3311	Fuel D 60/40 isooctane / toluene 5.69g, 0.5" squares	20	20	20	<0.5	<0.5	<0.5
CL11-3311	Fuel D 60/40 isooctane / toluene 6.00g, 1.0" squares	24	24	24	<0.5	<0.5	<0.5

6.8 MATERIAL COMPATIBILITY

Material compatibility testing was never envisioned as part of the original scope of this effort. However, after discussions with the Army, it was determined that the effect on the material is as important as the effect on the fuel. In an indirect way, ASTM D381 can also be an indicator of the severity of change that a material is undergoing after having been exposed to a fuel. Extraction of plasticizer will cause embrittlement in an elastomer leading to premature failure of the material. As it happened, another task under this work directive was already investigating material compatibility in tactical refueling systems and two of the materials being investigated were polyurethane and nitrile[2]. Based on O-ring testing, polyurethane exhibited some of the widest swings in physical properties after exposure to fuel blends; see polyurethane % change in volume (Figure 3) versus nitrile (Figure 4). There is no hard relationship between loss of plasticizer and material failure, but the ASTM D381 test in combination with other physical tests may provide an indication of a potential problem.

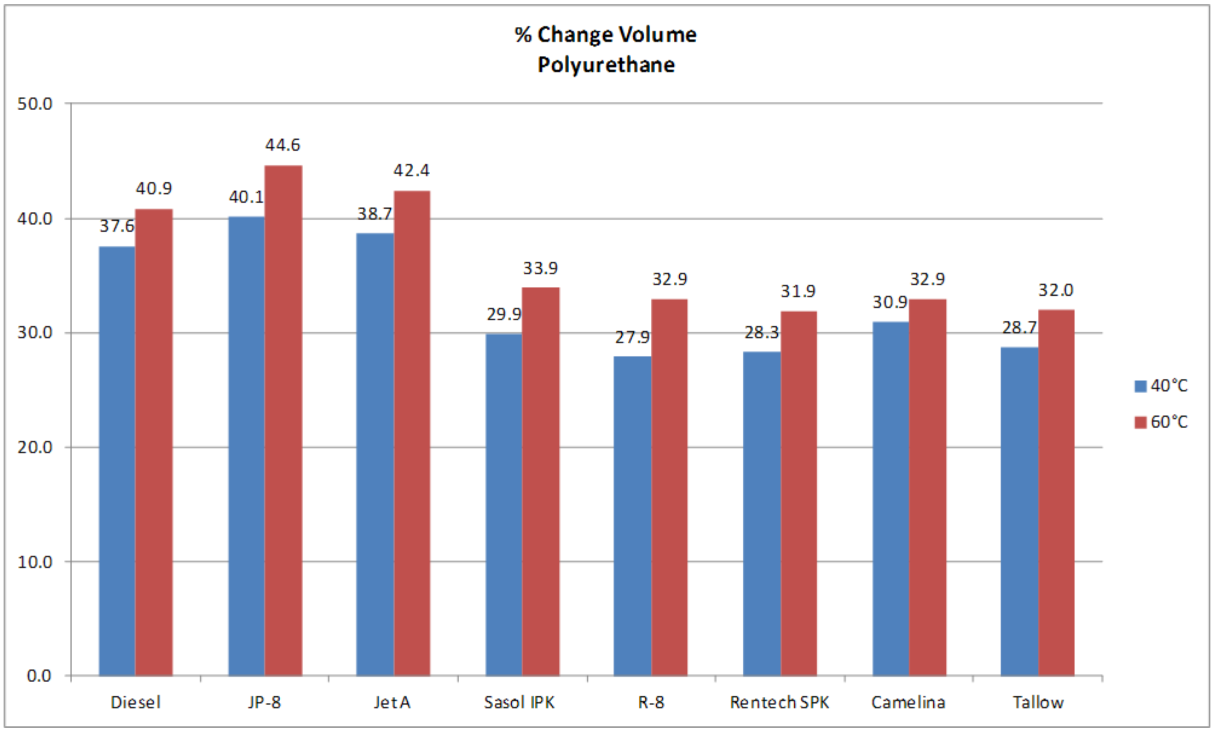

Figure 3. Percent Change in Volume Swell - Polyurethane.

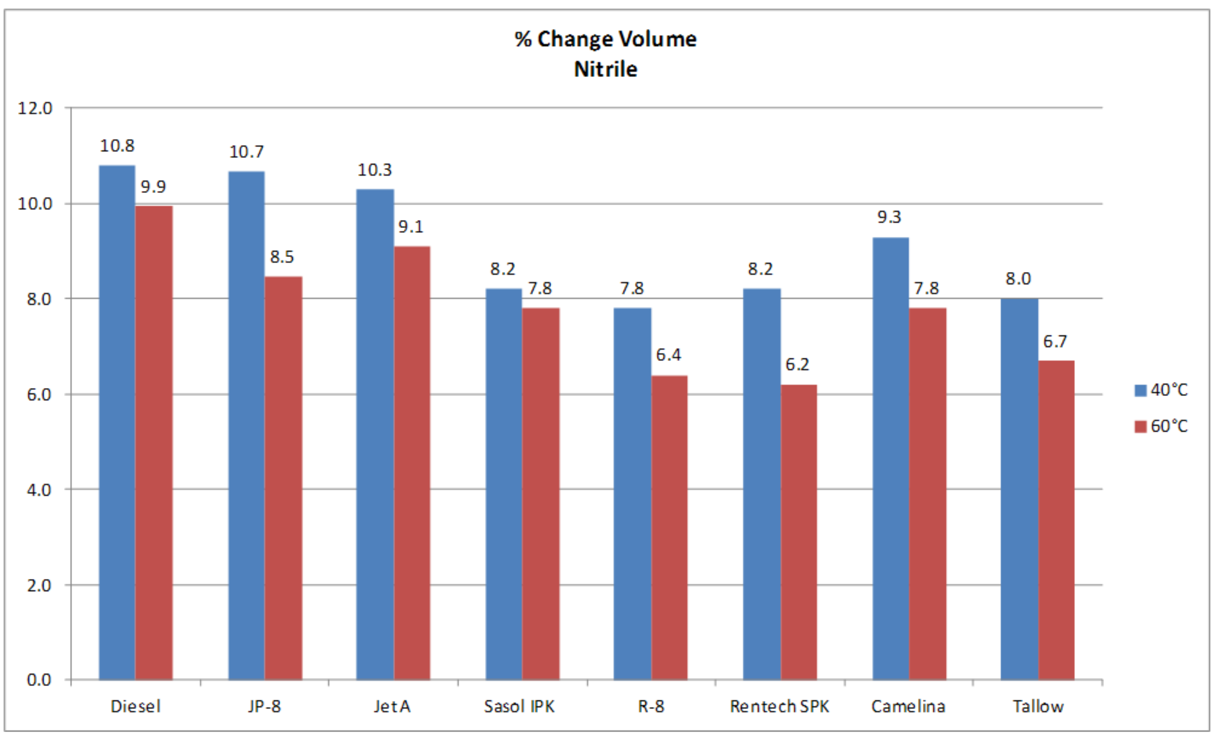

Figure 4. Percent Change in Volume Swell - Nitrile.

6.9 TERMINOLOGY

Both MIL-PRF-32233 and MIL-PRF-370J need to be updated because the terminology related to gum content is no longer accurate. The following excerpt from D381 indicates the changes to the terminology:

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Existent gum* - the evaporation residue of aviation fuels, without any further treatment

3.2 For non-aviation fuels, the following definitions apply:

3.3 S*olvent washed gum content*—the residue remaining when the evaporation residue (see 3.4) has been washed with heptane and the washings discarded.

3.3.1 *Discussion*—for motor gasoline or non-aviation gasoline, solvent washed gum content was previously referred to as existent gum.

3.4 *Unwashed gum content*—the evaporation residue of the product or component under test, without any further treatment.

Specific changes to each method are discussed below.

7.0 RECOMMENDED CHANGES

The following sections provide required and recommended changes to the current military specifications.

7.1 MIL-PRF-32233

The following are ***required*** changes to MIL-PRF-32233:

- Replace reference to “existent gum” with “solvent washed gum”
- Refer to “Unwashed gum” as “Existent/Unwashed gum”
- Remove “procedures 11.1 through 11.12” and leave out to prevent further confusion with later revisions of ASTM D381.
- Clarify which method, air or steam, to use for specific fluids.

- Update Table I:
  - Replace “Unwashed Gum” with “Existent/Unwashed Gum”
  - Replace “Existent Gum” with “Solvent Washed Gum”
- Update Table V:
  - Replace “Existent Gum” with “Solvent Washed Gum”
- Include a calculation to correct the measured values for the inherent gums in the test fluids.

The following are ***recommended*** changes to MIL-PRF-32233:

- Remove reference to “interior coating”

Suggested Modification:

4.5.2.2 Gum Content. Cut a 0.2-ounce specimen of each tank material into approximately 0.0625-inch squares and place in a flask containing 8.5 fluid ounces of Reference Fuel D of ASTM D471 and allow to stand for 48 hours at 73 ±5 °F. A sample of the fuel shall be retained for later use. Decant and filter the contaminated fluid through Whatman 41H filter paper or equal. Determine the existent/unwashed gum content of the filtrate in accordance with ASTM D381 using the air jet (gasolines and solvents) or steam jet (diesel and turbine fuels) vaporizing medium and an evaporation time of 45 minutes. Using the same samples after completing the existent/unwashed gum content test above, determine the solvent washed gum in accordance with ASTM D381. A minimum of three specimens shall be used. The retained fuel sample shall be tested with the same methods. The existent/unwashed gum content from the original fuel sample shall be subtracted from the existent/unwashed gum obtained from the fuel used in the soak test. The solvent washed gum from the original fuel sample shall be subtracted from the solvent washed gum obtained from the fuel used in the soak test. Nonconformance to Table I shall constitute failure of the test. This test shall be repeated with Reference Fuel B, Aviation Gasoline per ASTM D 910 Grade 100LL, JP-8, and DL2.

7.2 MIL-PRF-370J

The following are ***required*** changes to MIL-PRF-370J:

- Replace reference to “existent gum” with “solvent washed gum”
- Refer to “Unwashed gum” as “Existent/Unwashed gum”
- Remove “procedures 11.1 through 11.12” and leave out to prevent further confusion with later revisions of ASTM D381.
- Clarify which method, air or steam, to use for specific fluids.
- Update Table III:
    - Replace “Existent Gum” with “Solvent Washed Gum”

The following are ***recommended*** changes to MIL-PRF-32233:

- Include a filtration step after the soak period to remove large debris.

Suggested Modification:

> 4.3.6.6 <u>Existent gum</u>. A hose specimen 14-in. long shall be cut from the selected test item. One end of the hose shall be stoppered with a clean non-corrosive plug, and secured with a suitable clamp. The hose shall be filled to within 2 in. of the open end with reference fuel B as specified in ASTM D471, and plugged in a manner similar to the other end. A sample of the fuel shall be retained for later use. This hose section shall be stored in a vertical position for 168 hours at a temperature of 100 ± 4°F. At the end of each 24-hour period, the fuel shall be agitated for 5 minutes by moving the specimen from the vertical to the horizontal and back to the vertical position at a rate of 2 cycles per minute. On completion of the 168-hour period, the fuel shall be agitated, as before, for 5 minutes and immediately removed. The removed fuel shall be tested for solvent washed gum IAW the air-jet solvent wash method of ASTM D381. The retained fuel sample shall be tested at the same time and with the same method. The solvent washed gum from the original fuel sample shall be subtracted from the solvent washed gum obtained from the fuel removed from the test hose. Nonconformance to Table III shall constitute failure of this test.

8.0 CONCLUSIONS

Material compatibility testing is a fundamental necessity when verifying the performance of a new test article. In addition to physical measurements, such as tensile strength, gum content per ASTM D381 provides a means of determining how a material is being affected chemically which could lead to premature failure. The supporting data provided herein 1) demonstrates that the fuels are aggressively extracting heavy material from the elastomers/rubbers undoubtedly changing the nature of the material, 2) provides supporting evidence for required changes to the military specifications, and 3) shows that the alternative aviation fuels behave similarly to their petroleum-derived counterparts with respect to their affect on materials.

9.0 REFERENCES

1. Bessee, G.B.; Hutzler, S.A; Wilson, G.R., "Propulsion and Power Rapid Response Research and Development (R&D) Support. Delivery Order 0011: Analysis of Synthetic Aviation Fuels." AFRL report No. AFRL-RZ-WP-TR-2011-2084. April 2011.

2. O'Brien, S.; Hutzler, S.A., "Alternative Fuels Compatibility With Army Equipment Testing – Alternative Fuels Material Compatibility Analysis," Report Pending, Feb 2012.